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13. ABSTRACT (Maximum 200 words)

Research has been involved in exploring new ionization methods for atmospheric pressure detection. This has been geared towards enhanced sensitivity and selectivity for monitoring of very specific classes of compounds such as phosphoroorganic compounds, i.e. chemical warfare agent analogs, explosives analogs and small neurotransmitter compounds. The obvious orientation of the work has been towards developing new methods for monitoring various classes of compounds of direct interest to the Army. In order to enhance selectivity for target molecules against background in real detection environments, resonant two-photon ionization has been explored for various -NO_2 containing aromatics as well as other related species. In order to enhance the efficiency of ionization atmospheric pressure DC and RF glow discharge sources have been studies. These ionization sources have been used in combination with liquid injection for detection of explosives such as HMX and RDX and various CWA analogs. In order to further enhance both the sensitivity and simplicity of

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Ion Mobility Spectrometry/Mass Spectrometry With Laser Produced Ions

Final Report

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1. Introduction

During the past funding period under ARO Grant #DAAL03-88-A-0191 we have been involved in exploring new ionization methods for atmospheric pressure detection. This has been geared towards enhanced sensitivity and selectivity for monitoring of very specific classes of compounds such as phosphoroorganic compounds, i.e. chemical warfare agent analogs, explosives analogs and small neurotransmitter compounds. The obvious orientation of the work has been towards developing new methods for monitoring various classes of compounds of direct interest to the Army. In order to enhance selectivity for target molecules against background in real detection environments, resonant two-photon ionization has been explored for various -NO_2 containing aromatics as well as other related species. In order to enhance the efficiency of ionization atmospheric pressure DC and RF glow discharge sources have been studied. These ionization sources have been used in combination with liquid injection for detection of explosives such as HMX and RDX and various CWA analogs. In order to further enhance both the sensitivity and simplicity of detection, the API glow discharge has been interfaced to a sweep slit TOF mass spectrometer. In our most recent work an ion trap/TOF device is being tested as a means of storing ions for very high sensitivity detection.

A Statement of Problem

The goal of this work was to study novel methods of ionization for enhancing sensitivity and selectivity for atmospheric pressure ionization detection. This included:

- (1) A study of laser multiphoton ionization of -NO_2 containing compounds under atmospheric pressure conditions as compared to under vacuum.
- (2) The development of a novel atmospheric pressure DC glow discharge source in helium.
- (3) The development of a novel atmospheric pressure RF glow source in helium or argon.

- (4) The development of a new atmospheric pressure ionization/time-of-flight mass spectrometer.
- (5) The development of a new ion trap/time-of-flight device.

Extensive work has been performed on items 1, 2 and 4, while preliminary results have been obtained on items 3 and 5.

B. Summary of the Most Important Results

1. Development of an Atmospheric Pressure DC Glow Discharge Source in He at 1 atm.

In our initial work, a novel atmospheric-pressure glow discharge source was explored. This discharge operates in the 1 kV range at typically 50-250 μ A current in atmospheric-pressure helium. The characteristic I vs. V curve for a glow discharge is observed, and the discharge can operate in the positive or negative modes. The background ions observed in the positive mode from the atmospheric-pressure ionization (API) cell in the mass spectrometer are characteristic of water clusters from background water in the cell, and almost no helium background ions are detected. This atmospheric-pressure glow discharge source was used to ionize various small biologicals, drugs, peptides, and explosives analogs that were introduced into the API cell via a direct insertion probe. The resulting mass spectra exhibit generally the MH^+ with minimal or no fragmentation in the positive mode, indicating that ionization of the analyte occurs softly through proton transfer from water clusters. Detection limits in the low-pg range were obtained in both the positive and negative modes. In addition, laser desorption was interfaced to this glow source in order to assist in volatilization of explosives and other high melting point compounds.

In further work on this glow source, we have continued to explore its capabilities for efficient atmospheric pressure ionization. In order to enhance sensitivity, we have studied the upper limits at

COCAINE HCl

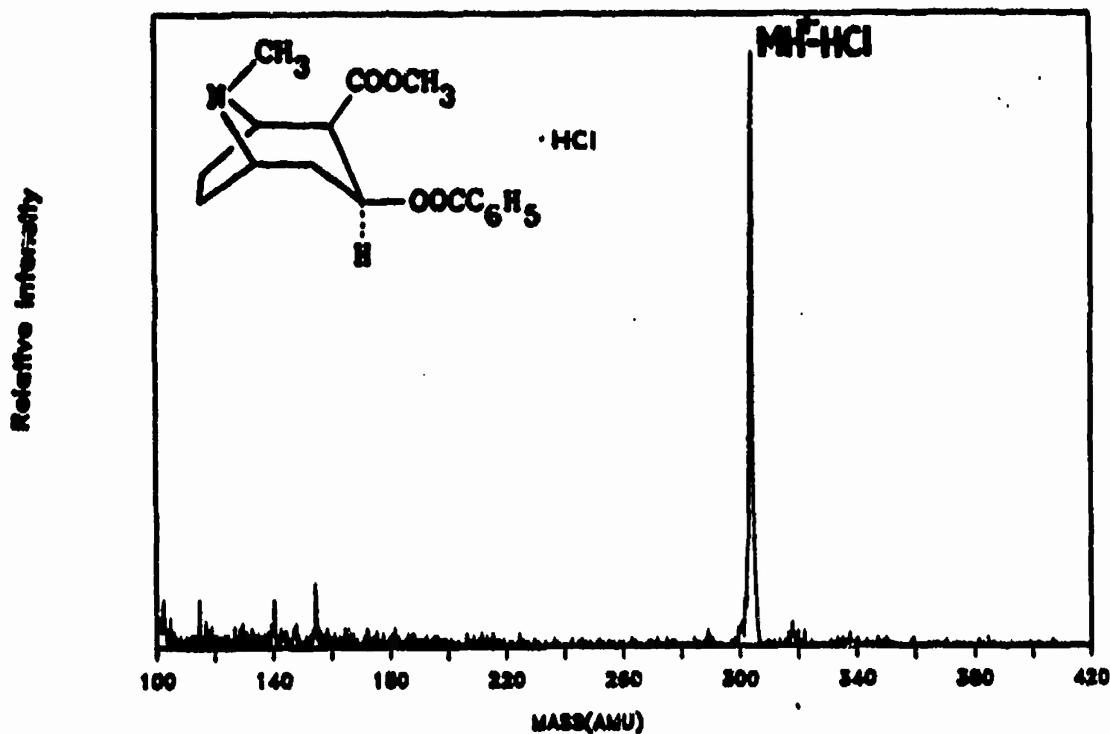


Figure 1: API glow discharge MS of methadone at 1 atm helium at 210°C.

Sensitivity Testing on Tyramine

glow discharge

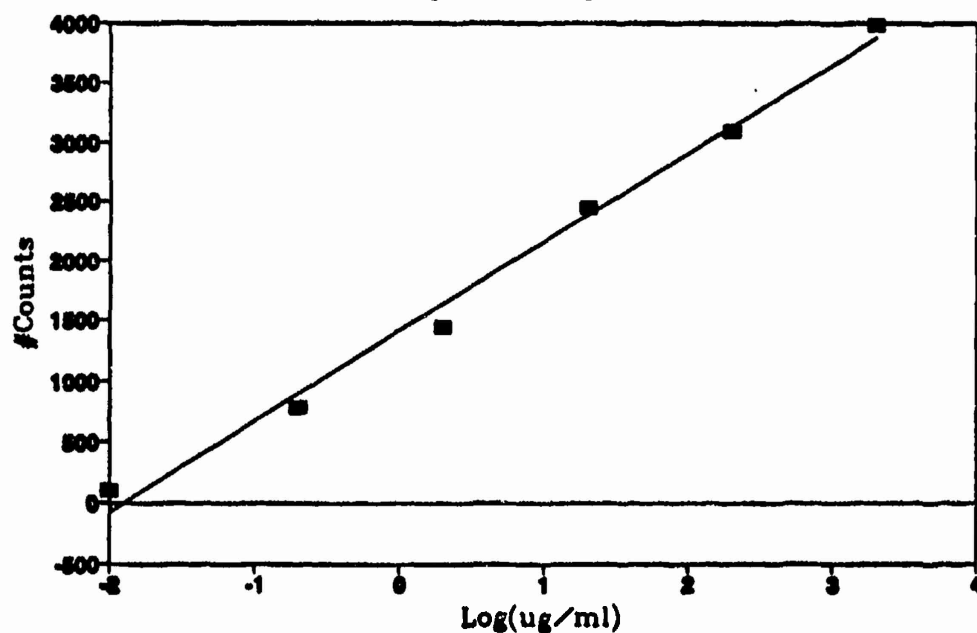


Figure 2: Quantitation curve for tyramine injected in methanol solvent into liquid nebulization/API glow discharge source.

which the glow can operate. In recent work we have been able to run the glow source in a stable manner up to 10 mamps of current, which is very comparable to the glow discharge under vacuum conditions. However, even under these conditions at atmospheric pressure, soft ionization of organic compounds is observed. We are able to sustain relatively high currents without arcing by adjusting the flow rate of the gas. Ionization by this source involves ionization of He or metastable formation, followed by proton transfer involving water clusters to the trace organic analyte. The key is that because of the high currents relative to corona sources, there are high densities of ions produced for API and these reactants ions are difficult to saturate. Thus one should be able to achieve very high sensitivity at the low end, and detection at the high concentration end without severe saturation as compared to the corona or Ni β source. In addition the water cluster reactions as a function of temperature and voltage were studied in the positive and negative modes.

In recent work we have interfaced a liquid capillary injection source which can inject liquid at up to 100 μ l/min into the glow. We have studied compounds including CWA analogs, explosives, drugs, phosphoroorganic pesticides, amino acids, oligopeptides, etc. (see Figure 1). A manuscript has been prepared on this work, but has not yet been submitted while we yet work to lower our detection limits. We can presently detect the molecule tyramine over a linear region of 6 orders of magnitude (see Figure 2). The lower limit of detection is 1.8 pg or <10 femtomole. On the upper end the ion molecule reactions are not easily saturated as in the corona source. This extended dynamic range is very important for real atmospheric monitoring, especially in battlefield conditions where other reactants may saturate the API source. Further instrumental improvements on our atmosphere-vacuum interface are presently in progress to enhance sensitivity. However, using this liquid injection methodology even HMX can be detected down to the pg range.

2. A major effort of this research has been to interface the DC glow source described above to a time-of-flight mass spectrometer.

The main problem here is that TOF devices require a start pulse to obtain time resolution and the source here is a continuous ion source. The device designed by our group utilizes a beam sweep method to generate a pulsed ion beam from a slit near the ion source for analysis by the TOF. In the past, API methodology has generally been interfaced to scanning mass spectrometers such as quadrupoles or magnetic sectors. However, TOF devices have several distinct advantages which include the ability to measure a full mass spectrum on every injection pulse and the ability to measure high mass with high transmission. In addition, TOF devices are relatively simple and inexpensive and could be designed for field monitoring purposes. In addition, TOF devices can achieve a resolution of 1000 or greater in combination with jet introduction.

In the present study, the high current atmospheric glow discharge source in helium was used to ionize various molecules of relatively low volatility injected into the API source via liquid injection. The resulting organic ions were introduced into a TOF, where using a beam modulation technique, pulsed ion packets were generated which could be rapidly mass analyzed in the TOF. The beam sweep-modulation technique was modeled and optimized for the various experimental parameters. A resolution of at least 519 at m/z 311 was obtained which was limited mainly by the experimental parameters in our present set-up. It is shown that as the mass increases, these experimental constraints become less important, and the resolution will continue to increase, limited mainly by the sweep speed across the beam modulation slit.

In figures 3 and 4 are shown the experimental set-up used in this work. It consists of a differentially pumped API-TOF mass spectrometer (Fig. 3) and a liquid injection sample source (Fig. 4). The liquid injection source delivers the sample dissolved in a solvent

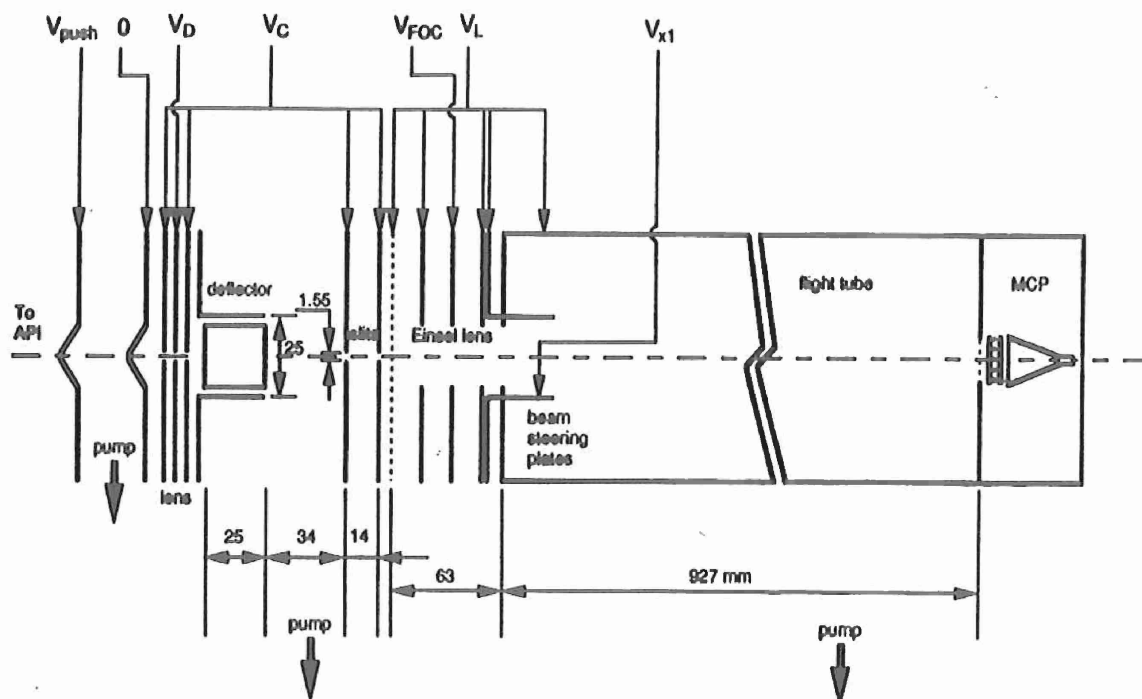


Figure 3: API/TOF set-up.

API INTERFACE

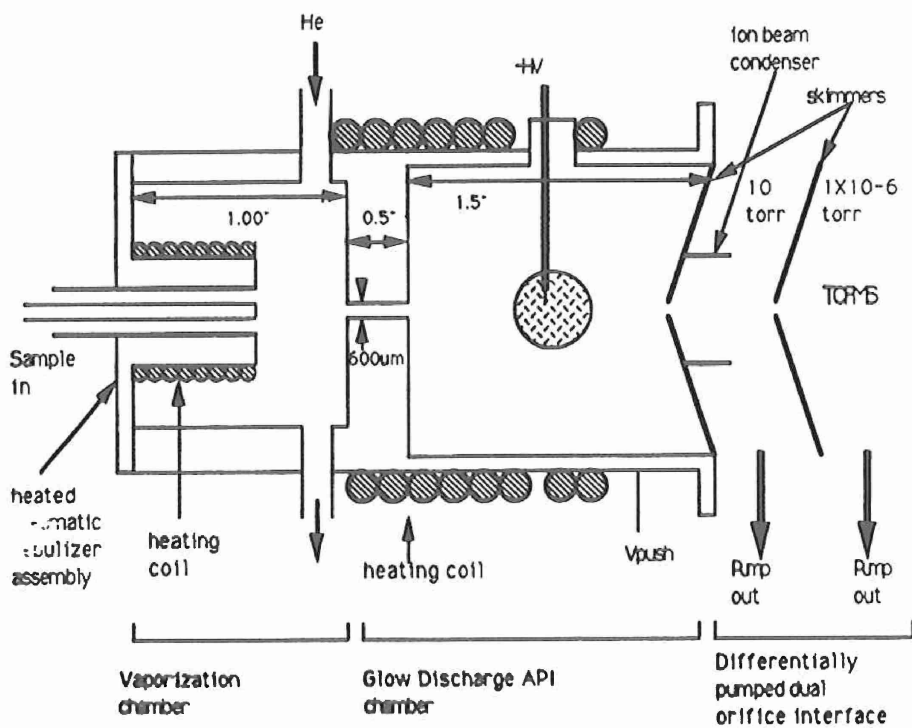


Figure 4: Liquid Nebulization/Vaporization Set-up.

through a heated pneumatic nebulizer assembly to the vaporization chamber where the sample is vaporized and the solvent removed. The sample then passes through a duct into a separate second chamber and is ionized via a glow discharge source in 1 atm He. The resulting ions are injected through a pair of differentially pumped skimmers, which sample the on-axis component of the ion beam. The ions are transported in a jet flow of He carrier which narrows the translational energy spread of the injected ions. The ion beam passes through several focusing slits and is then swept across a collimating slit using a rectangular sweep motion via four beam deflection plates. This beam sweep triggers the start pulse for the TOF mass analysis and also determines the mass resolution of the instrument. The slit width and the speed with which the ion beam is swept across the slit determine both the throughput and the size of the ion packet introduced into the flight tube. The ion packet enters a field free drift region ~ 1 m long which is pumped to a pressure of 3×10^{-7} torr using a 4" diffusion pump. These ions are detected at a triple microchannel plate detector.

Some representative API/TOF mass spectra are shown in figures 5 and 6. For example, 3-bromoaniline (Figure 5) was injected from liquid methanol into the heated nebulization chamber at 175°C. A protonated molecule parent ion is observed at m/z 172 and 174 with no accompanying fragmentation. The two ion peaks correspond to the two isotopic peaks of Br, i.e. ^{79}Br and ^{81}Br . These two isotopes are present in a natural abundance of 50.5:49.5 and thus two almost equal peaks are observed in the TOF mass spectrum. The two peaks are separated almost to the baseline, limited by the ^{13}C isotopic peak of the 3- ^{79}Br bromoaniline at m/z 173. The FWHM peak width in this case is approximately 40-45 ns. Thus a resolution of nearly 440 is obtained in this spectrum. In Figure 6 is shown the API/TOF mass spectrum of the drug amitriptyline injected from liquid methanol at 225°C. Only a protonated molecule peak, MH^+ , is observed at m/z 279 with no accompanying fragmentation. The peak at m/z 280 is the ^{13}C isotope peak which is present at about 20% of the amitriptyline parent ion. The isotopic peak is clearly resolved and the FWHM of the ion peaks

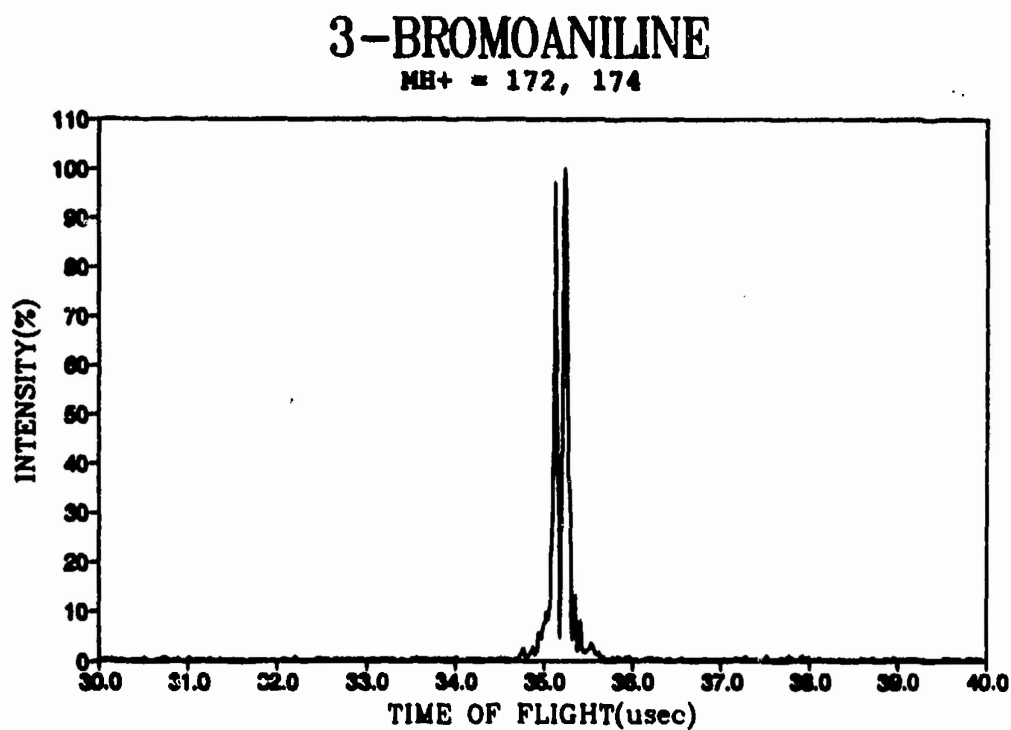


Figure 5: API/TOF mass spectrum of 3-bromoaniline using the glow discharge source in He.

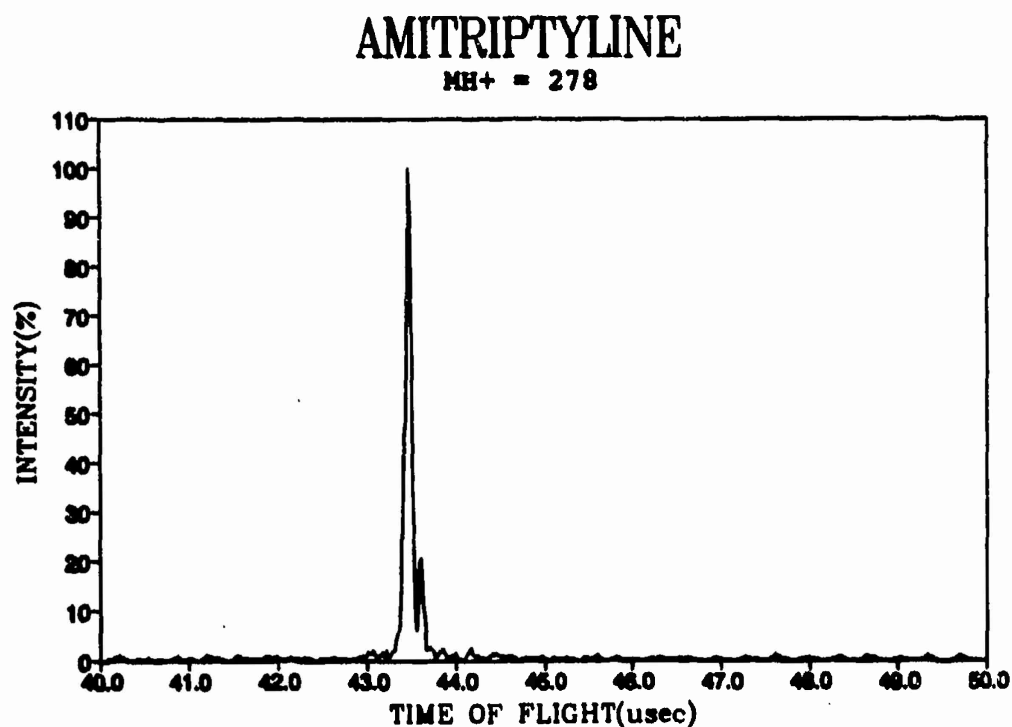


Figure 6: API/TOF mass spectrum of Amitriptyline using the glow discharge source in He.

are around 40-50 ns FWHM. Thus a resolution of ~ 435 is obtained in this spectrum. The sensitivity for the API/TOF is typically in the low ng range for these compounds and detection limits down to 1 pg have been achieved for an aniline sample. Thus, reasonably good resolution and high sensitivity can be obtained using the rather simple API/TOF. The results obtained are comparable to or better than that obtained by quadrupoles or other portable MS devices. We have also explored the coupling of electrospray introduction to an API/TOF device. Some results have been obtained for detection of molecules of $m/z < 1000$. We are presently trying to improve the detection limits of this device, which are not yet comparable to an API quadrupole. Our group has recently developed a triple channelplate detector with a gain of 10^8 - 10^9 X in order to enhance the sensitivity. The sensitivities typically reach down to < 100 pmole, but not yet down to the < 10 pmole level for this electrospray device.

3. The development of an ion trap/time-of-flight device.

Our group has been involved in developing new MS detection tools for API detection, i.e. for ion mobility. It is essential that the method remain relatively simple and retain high sensitivity. The device we are now developing is an ion trap/time-of-flight combination. The key here is that an RF ion trap is very simple to build, requires only a simple radio RF power supply at 1.1 MHz and can operate very well at relatively high pressure (i.e. 1-10 mtorr). The ion trap can act as an ion storage device to store and integrate ions for a long period of time for enhanced sensitivity. In order to analyze the ions the RF voltage is usually scanned and as each mass becomes unstable is ejected from the trap to the detector. In our set-up, we will make all ions stored in the device unstable together using a high voltage DC pulse on the end cap electrode. The ejected ions will be mass analyzed and detected in a simple linear time-of-flight MS. The TOF device first requires simple DC voltages (no current) and so the combination can be made relatively compact and simple. In addition, the ion trap operates at high pressure and so is excellent for API introduction.

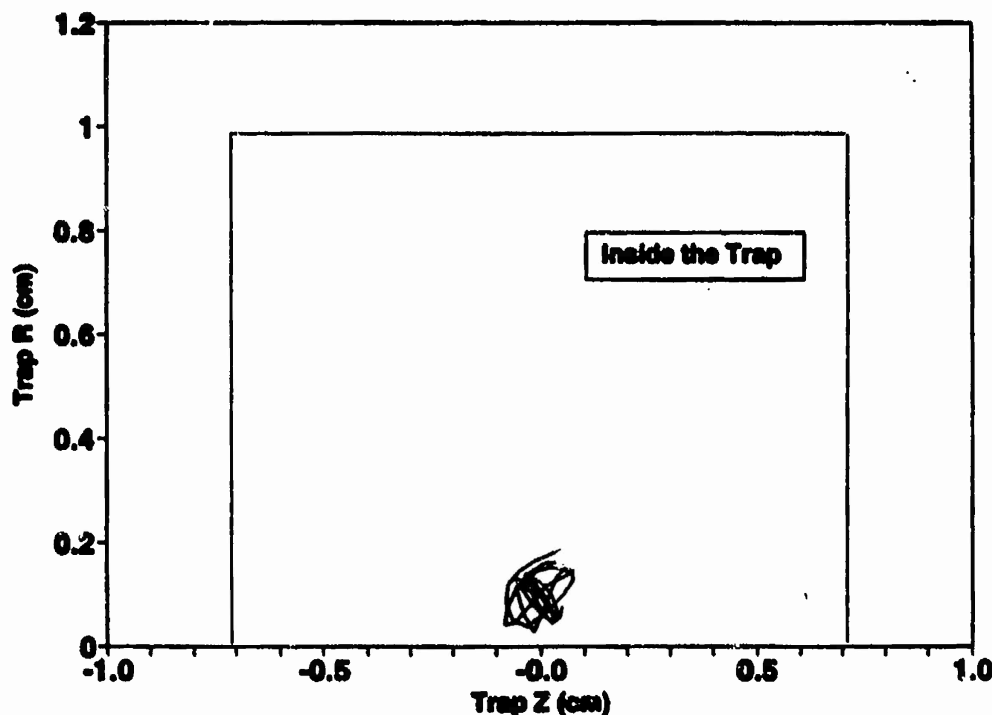


Figure 7: Ion trajectory in the ion trap space for the conditions $m/z = 570$ u, $KE = 0.1$ eV, $RF V = 1$ kV.

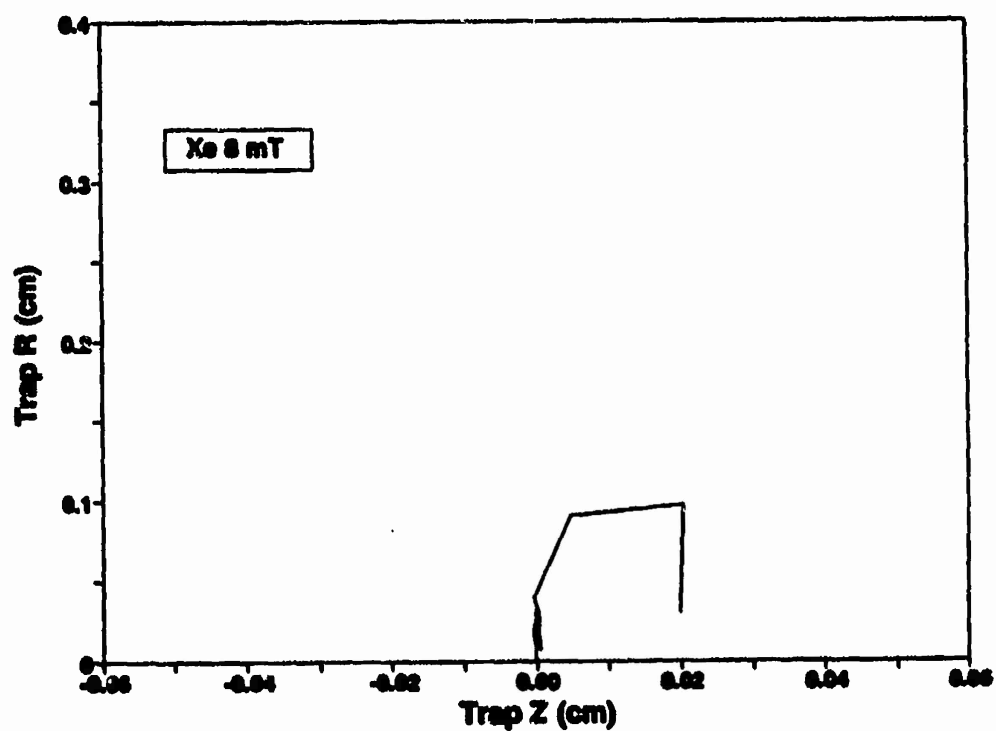
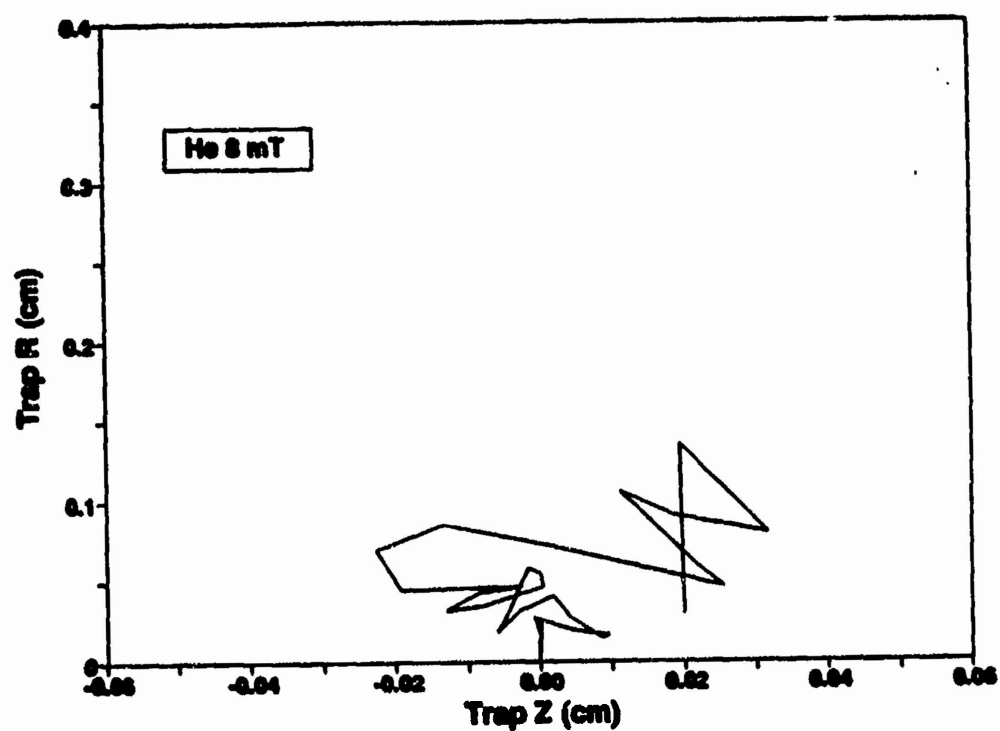
In initial work we have modeled the ion trap device on a PC computer. The computer simulation of the motion of ions in a three-dimensional (3-D) quadrupole ion trap were performed using a CompuAdd 286 PC/AT computer. The SIMION program was used as the main program to calculate the potential array of the ion trap space. Several user-written programs were interfaced to the SIMION program to simulate the effects of changing various operating conditions, such as the radio frequency (RF) potential, the collisional buffer gas, external ion injection and ejection from the trap. Using this simulation, the total storage mass range could be obtained as a function of RF voltage. The study demonstrated that the collisional buffer gas plays an important role in both stabilizing the trajectory of high kinetic energy ions (hot ions) inside the ion trap and trapping ions injected from an external source. The total mass ejection that results from applying a DC pulse on the output end cap electrode was

also studied. Some of the conclusions that resulted from this simulation can be summarized as follows:

In these simulations the trap was operated with an RF voltage on the ring electrode while the end caps remained at ground with no DC voltage applied to either electrode ($U=0$). It was shown that ions at relatively low KE, 0.1 eV, could be trapped effectively for a given rf voltage over a mass range limited by $(m/z)_{\min}$ and $(m/z)_{\max}$. As the rf voltage increases the mass range of the ions that can be stored stably in the trap increases. This increase in rf voltage is also accompanied by a higher $(m/z)_{\min}$ than can be stored in the trap. A typical stable trajectory for an ion of (m/z) 570 u at an rf voltage of 1 kV is shown in Figure 7.

It was further demonstrated in our simulations that as the KE of the ions increase above 50 eV, it becomes increasingly difficult to store the ions in the trap. However, if a buffer gas such as He is added to the trap, ions of even 50 eV energy can be stored in the trap over an extended mass range. At $P=1$ mTorr, ions of $(m/z) > 8 \times 10^5$ u can, in theory, be stored in the trap with an RF voltage of 10 kV. This will be of great significance in the use of traps for sequencing of proteins and DNA. Further the effect of different buffer gases, i.e. He, Ar, Kr and Xe were studied. It was found that buffer gases of increasing m/z were increasingly effective in damping the motion of an ion in the trap so that the ion motion tended towards the center of the trap. This is shown in Figures 8a and 8b for the use of He vs. Xe as a buffer gas.

Ions injected from external sources into the trap were simulated. Ions of > 20 eV could be effectively trapped using a He buffer gas and the motion of the ion was rapidly dampened to the center of the trap within 50 μ s. We also studied injection of ions of higher energy into the trap and showed that ions of even 200 eV can in theory be effectively injected into the trap from an external source if a buffer gas is present and simulations showing the damping of the ion motion in the trap have been demonstrated. In these simulations



Figures 8a and 8b: Simulated ion trajectories in (a) He, (b) Xe, where $m/z = 2000$, in initial KE = 50 eV, pressure = 8 mTorr.

it was shown that trapping of externally injected ions could be aided by introduction of the ions at an angle of 1° to the z-axis. If no buffer gas is used, then external ion ejection can be performed only for ions of very low energy, 4-15 V, and only if the injection is performed off the z-axis, where the ions can experience the effect of a greater applied force.

A unique demonstration in this work is the effect of a rapidly applied DC pulse to the end cap. The DC pulsed voltage causes the trajectory of the ions in the trap to become unstable and rapidly ejects the ions from the trap. The simulation of this phenomenon shows that ions of high (m/z) take longer to be ejected from the trap than ions of low (m/z). In addition, an increasing DC voltage ejects ions of a given (m/z) at an increasingly faster rate. Ions are usually ejected from ion traps by scanning the RF voltage. However, by placing a DC pulse on the end caps, the ion trap can be used as a storage device and the ions ejected for analysis by another device with different characteristics from the ITMS, i.e. a sector instrument, time-of-flight, or fourier transform mass spectrometer. The ability to inject ions efficiently into a TOFMS, for example, with time resolution will depend upon the characteristics with which ions are ejected by the DC pulse.

In our most recent work we have constructed an ion trap/time-of-flight combination. The ion trap has worked well in initial tests using a rapid RF voltage and electron impact ionization (EI). In addition, the idea of pulsing ions out of the trap using a high voltage DC pulse has worked very efficiently, although resolution has not yet been obtained in the TOF. The work on this aspect of the project is presently in progress.

4. The development of a novel atmospheric pressure RF glow discharge source.

Only preliminary work has been performed on the use of an RF glow source. The RF glow can be sustained in 1 atm. of argon or helium. In order to maximize the sensitivity of the source a long

discharge tube (~ 10 cm) was used from the discharge source to the orifice plate mode. In initial results, organic molecules such as aniline and methylnaphthalene were studied. Soft ionization can be observed depending upon the exact conditions, i.e. rf frequency, voltage, etc. The ionization appears very efficient and this method will continue to be studied in our laboratory.

5. A study of the MPI of -NO_2 containing compounds under atmospheric pressure.

One of our key studies has been the detection of -NO_2 containing explosive analogs at atmospheric pressure using UV laser-induced multiphoton ionization. Laser MPI has been studied quite extensively for aromatic compounds and azabenzenes with electron-repelling groups; however, almost no work has been performed on laser MPI of compounds with electron withdrawing groups containing -NO_2 , -COOH or -CN . We have focused on the study of -NO_2 containing compounds since these are analogs of explosive compounds. In particular, laser MPI of p- and o-nitrotoluene, nitrophenol, nitroaniline, and nitroanisole at 266 nm has been studied. A 266 nm photon (= 9.32 eV in a 2 photon process) is sufficient to ionize these compounds in a two-photon process except for the nitrotoluenes. Nevertheless, even the latter ionize readily at 266 nm.

In particular, R2PI at 1 atm in He and N_2 has been studied as a selective and specific method for softly ionizing of fragmenting substituted nitrobenzenes. R2PI at 266 nm in 1 atm He is shown to produce relatively soft ionization where either the molecular ion, M^+ , is observed or a specific fragment ion such as $(\text{M-NO})^+$ or $(\text{M-OH})^+$. In the case of P-nitrophenol at 266 nm the dominant peak is $(\text{M-OH})^+$ ($m/z = 122$) while in P-nitroaniline, the dominant peak is $(\text{M-NO})^+$ ($m/z = 108$) (see Figure 9b). In the case of P-nitroanisole, the dominant peak is also $(\text{M-NO})^+$. As the laser power is increased, additional fragments may be produced; however, ions due to fragmentation of the aromatic ring are rarely observed even at the

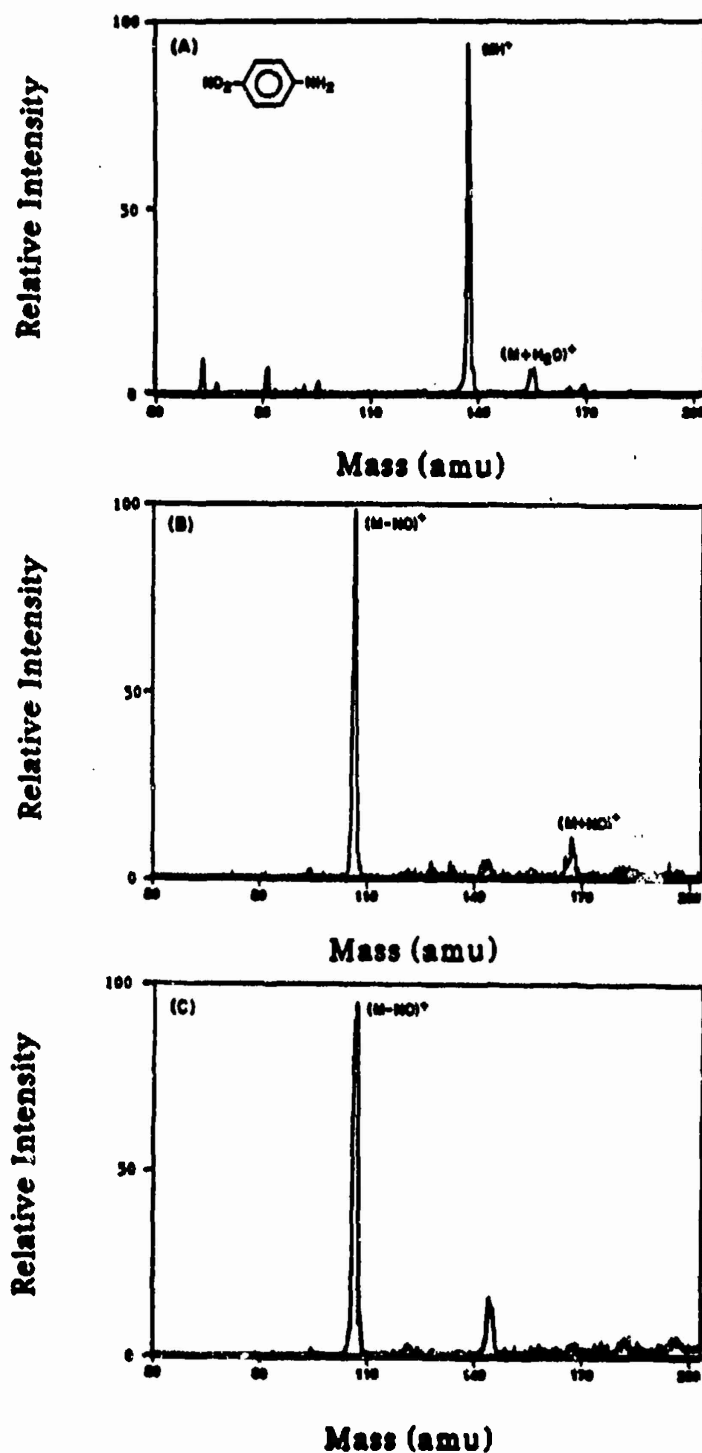


Figure 9: API/MS of p-nitroaniline at 140°C in helium obtained by (a) glow discharge source, (b) laser-induced R2PI at 266 nm and (c) vacuum-UV photoionization at 10.0 eV.

highest laser powers used ($> 10^9$ W/cm²). This contrasts to analog studies performed under vacuum at 10^{-6} torr where extensive fragmentation, including CH_n^+ could be observed at an equivalent laser power. The idea of atmospheric pressure collisional deactivation was clearly shown in these studies. Even at reduced laser power, though, the molecular ion was never observed in either the API or vacuum studies.

It was also shown in these studies that the ions produced by R2PI depend on the laser photon energy, and it was demonstrated that by selecting specific frequencies of light, that specific ions can be enhanced in the mass spectrum of a given molecule. It was also shown that under the same conditions distinctly unique ions are produced for the para, meta, and ortho isomers of p-nitroaniline, thus allowing easy discrimination among the isomers. Also the ion products obtained under atmospheric pressure conditions are shown to be uniquely different than those obtained by conventional methods such as electron impact or chemical ionization under vacuum conditions.

In order to further study the ionization process at atmospheric pressure, VUV lamps were used to directly photoionize substituted nitrobenzenes. The sum of two 266 nm photons corresponds to 9.32 eV. Thus, 9.2 eV and 10 eV lamps were used to study these compounds in atmospheric pressure helium. The results observed were very similar to those obtained by R2PI. Using both the 9.2 eV and 10.0 eV lamps, the dominant ion peak for p-nitroaniline was $(\text{M}-\text{NO})^+$ (see Figure 9c). Thus, the lack of molecular ion, M^+ , is probably due to ionization followed by rapid dissociation rather than by predissociation followed by ionization. Similar results were obtained for other substituted nitrobenzenes, where the ions produced via direct photoionization were very similar to those produced by R2PI at 266 nm. In our most recent work, a BBO crystal has been used to generate radiation at 213 nm. Using this wavelength, we have successfully ionized p-nitrotoluene and 2,6 DNT. In the case of 2,6 DNT we observe a single characteristic $(\text{M}-\text{NO})^+$ ion with no additional fragmentation. The 10.0 eV lamp proved to be of insufficient energy

to ionize 2,6 DNT. As of yet, we have been unable to successfully ionize 2,4,6 TNT at 213 nm.

C. List of Publications

1. An Atmospheric-Pressure Glow Discharge Ionization Source. I. Sofer, J. Zhu, H. S. Lee, W. Antos, D. M. Lubman. Appl. Spec. 1990, 44, 1391-1398.
2. Selective Laser-Induced Resonant Two-Photon Ionization and Fragmentation of Substituted Nitrobenzenes at Atmospheric Pressure. J. Zhu, D. Lustig, I. Sofer, D. M. Lubman. Anal. Chem. 1990, 62, 2225.
3. Laser-Induced Resonant Two-Photon Ionization of -NO₂ Containing Aromatic Compounds at Atmospheric Pressure. I. Sofer and D. M. Lubman, Proceedings of the 1989 Conference on Chemical Defense, Aberdeen Proving Ground, MD, 1990, p. 359.
4. The Design of an Atmospheric Pressure Ionization/Time-of-Flight Mass Spectrometer Using a Beam Deflection Method, C. Ma, S. M. Michael, M. Chien, J. Zhu and D. M. Lubman. Rev. Sci. Instrum., in press.
5. Computer Simulation of the Operation of a Three-Dimensional Quadrupole Ion Trap, C. Ma and D. M. Lubman, submitted.
6. High Pressure Liquid Injection of Biologicals into an Atmospheric Pressure DC Glow Discharge. J. Zhao and D. M. Lubman, submitted.

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S. Michael - Graduate Student - M. A. (currently Ph.D. student)
H. S. Lee - Graduate Student - M. A.
W. Antos - Graduate Student - M. A.